

JC07 Rec'd PCT/PTO 30 OCT 2001

FORM PTO-1390 U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE (REV. 10-95)		ATTORNEY'S DOCKET NUMBER  C3913(C)
TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. § 371		U.S. APPLICATION NO. (If known, see 37 C.F.R. § 1.7) <b>10/009810</b>
INTERNATIONAL APPLICATION NO.  PCT/EP00/03724	INTERNATIONAL FILING DATE  20 APRIL 2000	PRIORITY DATE CLAIMED  30 APRIL 1999
TITLE OF INVENTION  CONCENTRATED PERFUME COMPOSITIONS AND MANUFACTURE OF FABRIC SOFTENING COMPOSITIONS THEREFROM		
APPLICANT(S) FOR DO/EO/US  FRASER, STUART BERNARD		
Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:		
<ol style="list-style-type: none"> <li>1. <input checked="" type="checkbox"/> This is a FIRST submission of items concerning a filing under 35 U.S.C. § 371.</li> <li>2. <input type="checkbox"/> This is a SECOND or SUBSEQUENT submission of items concerning a filing under 35 U.S.C. § 371.</li> <li>3. <input type="checkbox"/> This express request to begin national examination procedures (35 U.S.C. §371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. §371(b) and PCT Articles 22 and 39(I).</li> <li>4. <input checked="" type="checkbox"/> A proper DEMAND for International Preliminary Examination was made by the 19<sup>th</sup> month from the earliest claimed priority date.</li> <li>5. <input checked="" type="checkbox"/> A copy of the International Application as filed (35 U.S.C. §371(c)(2)) <ol style="list-style-type: none"> <li>a. <input type="checkbox"/> is transmitted herewith (required only if not transmitted by the International Bureau).</li> <li>b. <input checked="" type="checkbox"/> has been transmitted by the International Bureau.</li> <li>c. <input type="checkbox"/> is not required, as the application was filed in the United States Receiving Office (RO/US).</li> </ol> </li> <li>6. <input type="checkbox"/> A translation of the International Application into English (35 U.S.C. §371(c)(2)).</li> <li>7. <input checked="" type="checkbox"/> Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. §371(c)(3)) <ol style="list-style-type: none"> <li>a. <input type="checkbox"/> are transmitted herewith (required only if not transmitted by the International Bureau).</li> <li>b. <input type="checkbox"/> have been transmitted by the International Bureau.</li> <li>c. <input type="checkbox"/> have not been made, however, the time limit for making such amendments has NOT expired.</li> <li>d. <input checked="" type="checkbox"/> have not been made and will not be made.</li> </ol> </li> <li>8. <input type="checkbox"/> A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. §371(c)(3)).</li> <li>9. <input checked="" type="checkbox"/> An unexecuted oath or declaration of the inventor(s) (35 U.S.C. §371(c)(4)).</li> <li>10. <input type="checkbox"/> A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. §371(c)(5)).</li> </ol>		
<b>Items 11. To 16. Below concern document(s) or information included:</b>		
<ol style="list-style-type: none"> <li>11. <input type="checkbox"/> An Information Disclosure Statement under 37 C.F.R. §§ 1.97 and 1.98.</li> <li>12. <input type="checkbox"/> An assignment document for recording. A separate cover sheet in compliance with 37 C.F.R. §§3.28 and 3.31 is included.</li> <li>13. <input checked="" type="checkbox"/> A FIRST preliminary amendment.</li> <li><input type="checkbox"/> A SECOND or SUBSEQUENT preliminary amendment.</li> <li>14. <input type="checkbox"/> A substitute specification.</li> <li>15. <input type="checkbox"/> A change of power of attorney and/or address letter.</li> <li>16. <input type="checkbox"/> Other items or information:</li> </ol>		

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U.S. APPLICATION NO. (If known, see 37 CFR §1.5)	INTERNATIONAL APPLICATION NO.	ATTORNEY'S DOCKET NUMBER
10/009610	PCT/EP00/03724	C3913(C)

17. <input checked="" type="checkbox"/> The following fees are submitted:				CALCULATIONS PTO USE ONLY	
<p>BASIC NATIONAL FEE (37 CFR §1.492(a)(1)-(5)):</p> <p>Search Report has been prepared by the EPO or JPO \$890.00</p> <p>International preliminary examination fee paid to USPTO (37 CFR §1.482) \$710.00</p> <p>No international preliminary examination fee paid to USPTO (37 CFR §1.482) but international search fee paid to USPTO (37 CFR §1.445(a)(2)) \$</p> <p>Neither international preliminary examination fee (37 CFR §1.482) nor international search fee (37 CFR §1.445(a)(2)) paid to USPTO \$</p> <p>International preliminary examination fee paid to USPTO (37 CFR §1.482) and all Claims satisfied provisions of PCT article 33(2)-(4). \$</p>					
ENTER APPROPRIATE BASIC FEE AMOUNT =				\$890.00	
Surcharge of \$130.00 for furnishing the oath or declaration later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR §1.492(e)).					
CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE		
Total Claims	14 - 20 =		X \$18.00		
Independent Claims	2 - 3 =		X \$80.00		
MULTIPLE DEPENDENT CLAIM(S) (if applicable)			X \$270.00		
TOTAL OF ABOVE CALCULATIONS =				\$890.00	
Reduction of 1/2 for filing by small entity, if applicable. A Verified Small Entity Statement must also be filed (Note 37 C.F.R. §§ 1.9, 1.27, 1.28).					
SUBTOTAL =					
Processing fee of \$130.00 for furnishing the English translation later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 C.F.R. § 1.492(f)).					
TOTAL NATIONAL FEE =					
Fee for recording the enclosed assignment (37 C.F.R. § 1.21(h)). The assignment must be accompanied by an appropriate cover sheet ((37 C.F.R. §§ 3.28, 3.31). \$40.00 per property.					
TOTAL FEES ENCLOSED =				\$890.00	
				Amount to be refunded:	
				Charged.	

- a. ☐ A check in the amount of \_\_\_\_\_ to cover the above fees is enclosed.
- b. ☒ Please charge Deposit Account No. 12-1155 in the amount of \$890.00 to cover the above fees. Triplicate copies of this letter are enclosed.
- c. ☒ The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 12-1155. Triplicate copies of this letter are enclosed.

Customer Number:



00201

PATENT TRADEMARK OFFICE

NOTE: Where an appropriate time limit under 37 C.F.R. §§ 1.494 or 1.495 has not been met, a petition to revive (37 C.F.R. §§ 1.137(a) or (b)) must be filed and granted to restore the application to pending status.

Respectfully submitted,

*Edward A. Squillante, Jr.*  
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10/009610

PATENT  
#99-0276-UNI  
Case #C3913(C)

Express Mail Label No.: ET 506 464 969 US

**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**

Applicant: Fraser  
Deposited: October 30, 2001  
For: CONCENTRATED PERFUME COMPOSITIONS AND MANUFACTURE  
OF FABRIC SOFTENING COMPOSITIONS THEREFROM

Edgewater, New Jersey 07020  
October 30, 2001

**PRELIMINARY AMENDMENT**

Assistant Commissioner for Patents  
Washington, D.C. 20231

Sir:

With regard to the above-identified application filed concurrently herewith, please amend the following:

**In the Claims:**

Please enter the following amended claims:

4. (Amended) A composition according to claim 1 comprising 40-85 wt% perfume.
5. (Amended) A composition according to claim 1 wherein the perfume has a solubility in water of equal to, or less than 0.5g in 100 ml of water at 20°C.
6. (Amended) A composition according to claim 1 comprising 0.2 wt% to 1 wt% dye.

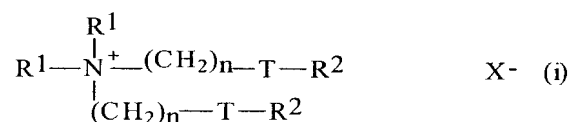
7. (Amended) A composition according to claim 1 wherein the dye has a solubility in water of equal to or greater than 5g of 100 ml of water at 20°C.

8. (Amended) A composition according to claim 1 comprising 10 wt% - 30 wt% cationic surfactant as the stabilising agent.

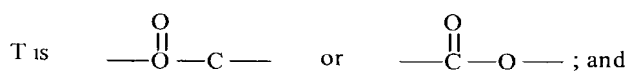
9. (Amended) A composition according to claim 1 wherein the cationic stabilising agent is a compound of general formula (A)



Wherein  $\text{R}^1$  and  $\text{R}^2$  are independently  $\text{C}_1\text{-C}_6$  alkyl, alkenyl, substituted alkyl or alkenyl groups, or hydroxyalkyl groups and  $\text{R}^3$  and  $\text{R}^4$  are independently  $\text{C}_8\text{-C}_{28}$  alkyl, alkenyl, substituted alkyl or alkenyl groups, or hydroxyalkyl groups or, a compound of general formula (I)



wherein each  $\text{R}^1$  group is independently selected from  $\text{C}_{1-4}$  alkyl, hydroxyalkyl or  $\text{C}_{2-4}$  alkyl groups; and wherein each  $\text{R}^2$  group is independently selected from  $\text{C}_{8-28}$  alkyl or alkenyl groups;  $\text{X}^-$  is chloride or methosulphate.



n is an integer from 0-5

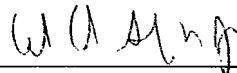


**REMARKS**

The present amendment is submitted to eliminate multiple dependencies and to correct minor typographical errors. The amendments were not intended to and should not be construed to have been made for any reasons related to patentability of the claims.

Attached hereto is a marked-up version of the changes made to the claims by the current amendment. The attachment is captioned "Version with Markings to Show Changes Made".

Respectfully submitted,




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Edward A. Squillante, Jr.  
Reg. No. 38,319  
Attorney for Applicant(s)

EAS/mt  
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$$\frac{d}{dt} \left( \frac{1}{2} m v^2 + U \right) = - \nabla \cdot (\mathbf{v} p) + \nabla \cdot (\mathbf{v} \cdot \boldsymbol{\tau})$$

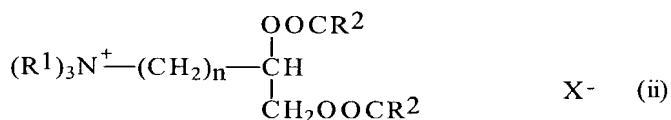
9. (Amended) A composition according to ~~any one of the preceding claims~~claim 1 wherein the cationic stabilising agent is a compound of general formula (A)



formula (I)

T is  $\begin{array}{c} \text{O} \\ \parallel \\ \text{---O---C---} \end{array}$  or  $\begin{array}{c} \text{O} \\ \parallel \\ \text{---C---O---} \end{array}$ ; and

or, a compound of general formula (ii)



10. (Amended) A composition according to ~~any one of the preceding claims~~claim 1 wherein the weight ratio of perfume to dye is within the range 200:1 to 5:1, preferably 100:1 to 15:1.



11. (Amended) A composition according to ~~any one of the preceding claims~~claim 1 wherein the weight ratio of perfume to stabilising agent is 10:1 to 1:1, preferably 5:1 to 1:1.

12. (Amended) A composition according to ~~any one of the preceding claims~~claim 1 comprising 0.1-10 wt% water.

13. (Amended) A method of preparing a fabric softening composition comprising the steps;

(i) preparing a base composition comprising a cationic and/or nonionic fabric softening agent, and

(ii) adding to (i) a composition according to ~~any one of the preceding claims~~claim 1,

to produce the fabric softening composition.

Concentrated Perfume Compositions and  
Manufacture of a Fabric Softening Compositions Therefrom

Technical Field

5

The present invention relates to concentrated perfume compositions. The invention further relates to a method of manufacturing a fabric softening composition from the concentrated perfume composition, and, to a fabric  
10 softening composition so produced.

Background and Prior Art

In the commercial manufacture of fabric softening  
15 compositions the accurate dosing of minor ingredients e.g. dyes, perfumes etc. is often problematic because of the low levels of these ingredients required in the compositions so that the relative proportions of these minor ingredients are difficult to control. Also when compositions are  
20 produced in batches this requires that the exact levels of minor ingredients, particularly dye and perfume, must be added individually which increases the possibility of inaccurate dosing.

25 It is also known that concentrated perfume compositions are prone to instability problems.

Thus there is a need to provide stable concentrated compositions of well-known minor ingredients such as dyes  
30 and perfumes for use in the manufacture of coloured and perfumed compositions such as fabric softening compositions.



- (a) 15 - 95 wt% lipophilic perfume,
- (b) 0.05 - 5 wt% water-soluble dye,
- (c) 4 - 50 wt% of stabilising agent comprising a  
cationic stabilising agent, and
- 5 (d) water-miscible solvent,

wherein the composition comprises between 0.1 to 20 wt%  
water, and the cationic stabilising agent has an  $L\alpha$  to  $L\beta$   
transition temperature of 45°C or below for a 5% wt  
10 dispersion of the stabilising agent in water, and the  
solvent is present in an amount of up to 10 wt%.

All percentages by weight herein refer to the percentage  
based on the total amount of the composition.

15

The invention also provides a method of preparing a fabric  
softening composition comprising the steps;

- (i) preparing a base composition comprising a  
20 cationic and/or nonionic fabric softening agent,  
and
- (ii) adding to (i) a composition as defined above  
to produce the fabric softening composition.

25 Furthermore the invention also provides a fabric softening  
composition obtainable by the method above.

The invention provides a concentrated perfume-containing  
composition that also contains dye, both in a much higher  
30 concentration than would typically be found in a fabric  
softening composition. This provides a stable pre-mix of  
minor ingredients (perfume and dye). Which can be prepared

at suitable ratios for direct dosage into a base composition. Excellent accuracy of the dosage of these minor ingredients into a base composition is achieved and this thus simplifies automated preparation of fabric softening compositions.

Furthermore the concentrated perfume and dye compositions allow accurate dosing of these minor ingredients to a base composition at a late stage (eg. after the main active ingredients have been mixed) of the manufacture of a fabric softening composition. This in turn allows a wide range of compositions to be prepared from a pre-compounded base composition, so providing easier and more versatile manufacture. Thus changing between variant formulations is simplified as only a small part of the automated production apparatus requires cleaning when the 'minors' are changed in the composition (as a single 'minor' composition can be used in the process). This provides reduced aqueous effluent, saves production time and provides increased production flexibility.

In particular, the invention provides the preparation of a concentrated composition of perfume and dye, wherein the perfume and dye are of different lipophilic and lipophobic characters.

#### Detailed Description of the Invention

The composition of the invention is preferably an isotropic liquid, most preferably a microemulsion, and especially a water-in-oil microemulsion.

It is preferred that if the composition is an isotropic liquid it does not contain liquid crystalline phases.

Where the compositions are not clear, they should be stable to storage at 20°C for several days eg. 2 days. Whilst some degree of cloudiness can be tolerated in the compositions, it is preferred that they are isotropic liquids. Such isotropic liquids may have included therein minor amounts of materials that are not isotropic, provided, the stability of the composition is not adversely affected.

#### Perfume

The perfume used in the invention is lipophilic in nature. By a lipophilic perfume is meant that the perfume has a solubility in water (i.e. it dissolves) of 1g or less in 100 ml of water at 20°C. Preferably the solubility in water is equal to or less than 0.5g, preferably equal to or less than 0.3g in 100ml of water at 20°C. Such perfumes may be referred to as water-insoluble perfumes.

20

The perfume may be any conventional perfume used in fabric softening compositions. The perfume will thus preferably be compatible with the fabric softening actives typically found in fabric softening compositions, although, not many commercially available perfumes will not be compatible. Also the perfume will generally be polar in nature.

When the composition is a water-in-oil microemulsion the perfume will, because of its lipophilic nature, form the predominant part of the oil phase. It is preferred if the perfume comprises 60% by weight or more, preferably 70% by

30

weight or more, of the oil phase when the composition is a water-in-oil microemulsion.

Perfumes contain a number of ingredients which may be  
5 natural products or extracts such as essential oils, absolutes, resinoids, resins etc. and synthetic perfume components such as hydrocarbons, alcohols, aldehydes, ketones ethers, acids, esters, acetals, ketals, nitriles, phenols, etc. including saturated and unsaturated  
10 compounds, aliphatic, alicyclic, heterocyclic and aromatic compounds. Examples of such perfume components are to be found in "Perfume and Flavour Chemicals" by Steffen Arctander (Library of Congress catalogue card no. 75-91398).

15

Any lipophilic perfume which is compatible with nonionic and/or cationic compounds may be used in the composition.

The compositions contain 15 - 95 wt% of lipophilic perfume,  
20 preferably 20 - 90 wt%, more preferably 25 - 85 wt%, such as 40 - 85 wt%, e.g. 45 - 80 wt%.

More than one lipophilic perfume may be used in the compositions of the invention.

25

#### Dye

The dye is an at least a sparingly water-soluble dye and may be any such dye conventionally used in softening and cleaning products. It is especially preferred that the dye  
30 has a solubility in water of equal to, or greater than, 2g in 100 ml of water at 20°C, preferably equal to or greater than 5g.

The dye may be an acid-dye or other suitable type of dye. For an acid dye, the solubility may be as high as 50g in 100ml of water of 20°C.

- 5 The dye is present in an amount of 0.05 - 5 wt%, preferably 0.1 - 2 wt%, more preferably 0.2 - 1 wt%, eg 0.25 - 0.7 wt%.

The weight ratio of perfume to dye is preferably within the  
10 range 200:1 to 5:1, more preferably 150:1 to 10:1, e.g. 100:1 to 15:1, such as 80:1 to 20:1. Depending upon the amount of dye required, the ratio could be towards the lower end of the ratio e.g. 40:1 to 25:1. However if only a  
15 e.g. 900:1 to 200:1, eg. 900:1 to 250:1.

#### Stabilising agent

The stabilising agent comprises a cationic stabilising agent having an  $L\alpha$  to  $L\beta$  transition temperature of 45°C or  
20 below for a 5 wt% dispersion of the stabilising agent in water. This  $L\alpha$  to  $L\beta$  transition can be measured by DSC as defined in "Handbook of Lipid Bilayers", D Marsh, CRC Press, Boca Raton, Florida, 1990 (pages 137 and 337).

- 25 Any cationic stabilising agent meeting the above transition temperature requirement, may be used according to the invention. Compounds falling within definitions of formulae (i) are (ii) below and meeting the transition temperature requirements are cationic stabilising agents according to  
30 the invention. Compounds falling within the definitions (i) and (ii) but which do meet the transition temperature

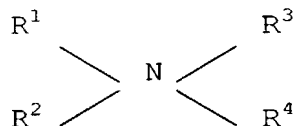


requirements are not cationic stabilising agents as herein defined.

The cationic stabilising agent used in the invention is preferably a cationic surfactant, more preferably one of the quaternary ammonium compounds of formulae (A), (i) or (ii) below. Compounds of these formulae are only stabilising agents as referred to herein if they meet the above transition temperature requirement.

10

(A)



15 wherein  $R^1$  and  $R^2$  are independently  $C_1$ - $C_6$  alkyl, alkenyl, substituted alkyl or alkenyl groups, or hydroxyalkyl groups and  $R^3$  and  $R^4$  are independently  $C_8$ - $C_{28}$  alkyl, alkenyl, substituted alkyl or alkenyl groups, or hydroxalkyl groups.

Preferably  $R^1$  and  $R^2$  are independently  $C_1$ - $C_2$  groups and  $R^3$  and  $R^4$  are independently  $C_{12}$ - $C_{22}$  groups,  $X^-$  is a compatible anion eg  $Cl^-$ ,  $MESO_4^-$ ,  $Br^-$ ,  $I^-$  acetate etc.

25

A cationic surfactant stabiliser according to formula (A) is ditallowyl dimethyl ammonium chloride.

The cationic stabiliser may also be a compound as defined by formulae (i) and (ii) below, provided, that compound meets the transition temperature requirements therein defined.

30

A cationic stabiliser according to formula (i) below is dioleyl ester of methyl triethanol ammonium methosulphate

wherein one  $R^1$  is methyl and one is hydroxyethyl, both n are 2, both T are O-C=O, and both  $R^2$  are tallow.

It is preferred that the stabilising agent is compatible  
5 with conventional fabric softening agents, and in particular with the fabric softening agents described herein. Mixtures of cationic stabilising agents may also be included. In many cases the cationic stabilising agent and the fabric softening agent will be the same compound.

10

Optional stabilising agent

Nonionic stabilising agents may be used in addition to the cationic stabilising agent. Preferably the nonionic stabilising agent is a nonionic surfactant. Suitable types  
15 of nonionic surfactants include alcohol alkoxyates especially ethoxyates, preferably  $C_8$ - $C_{20}$  alkyl esters alkoxyated with an average of 1 to 10 alkoxyate units, preferably 1 to 7 alkoxyate units. The ethoxyates, especially secondary alcohol ethoxyates, are particularly  
20 preferred.

The total amount of stabilising agent (cationic or cationic plus nonionic) in the compositions is 4 wt% - 50 wt%, preferably 10-30 wt%, more preferably 15-25 wt%. Where a  
25 mixture of cationic and nonionic stabilisers are used, the weight ratio of cationic:nonionic is preferably in the range 99:1 to 50:50, more preferably 99:1 to 60:40.

It is especially preferred that 10wt% - 30wt% cationic  
30 surfactant is used as the stabilising agent, plus optionally an additional amount of nonionic stabilising agent.

The weight ratio of perfume to the total amount of stabilising agent is preferably within the range 10:1 to 1:1, more preferably 8:1 to 1:1, e.g. 5:1 to 1:1, e.g. 3:1 to 1:1.

5

Water-miscible solvent

The compositions comprise water-miscible solvents in amounts of up to 10wt% of the composition, preferably up to 7.5 wt% as an essential element of the invention. The  
10 solvent is preferably present in an amount of 0.1 - 10wt %, more preferably 0.5 to 7.5 wt%.

The water-miscible solvent may be solvent having a C<sub>1</sub>-C<sub>6</sub> alkyl chain such as ethanol or isopropanol. The solvent may  
15 be present in the compositions either through direct addition or it may be added by being present in the stabilising agent or other component of the composition.

The compositions may also include low amounts (up to 5% by  
20 weight) of 'minor' ingredients (other than dye or perfume) typically found in fabric softening compositions, provided, the stability of the composition is not affected. 'Minor' ingredients that may be included include fatty acids, non-aqueous solvents, fluorescers, hydrotropes, antifoaming  
25 agents, anti-redeposition agents, enzymes, optical brightening agents, opacifiers, anti-shrinking agents, anti-wrinkle agents, anti-spotting agents, germicides, fungicides, anti-oxidants, UV absorbers (sunscreens), sequestrants, preservative, chlorine scavengers, pH  
30 buffering agents, dye fixatives, anti-corrosion agents, drape imparting agents, and antistatic agents.

The compositions comprise 0.1-20 wt% water, preferably 0.1-15 wt%, more preferably 0.1-10 wt%.

Any suitable method of preparing the compositions of the invention may be used. For example the perfume, stabilising agent and any other oil soluble ingredients are mixed together to form a clear liquid. Gentle heating and/or stirring may be necessary at this point to produce the clear liquid (typically heating at 25-45°C). Water-soluble ingredients including the dye (and other 'minor' ingredients such as preservative) are dissolved separately in the required amount of water. The water-soluble aqueous portion is added to the perfume containing mixture, in aliquots if required, with stirring to produce the composition.

A further aspect of the present invention provides a method of producing a fabric softening composition by adding a composition of the invention to a base composition comprising a cationic and/or nonionic fabric softening agent. Preferably the base composition is aqueous. The addition may be made in any suitable manner.

The composition is added at a suitable % by weight to give the required amount of perfume and dye etc. in the resultant fabric softening composition. The addition amount, and effectiveness of mixing, can be easily checked by measuring the colour. The fabric softener composition produced by the above method contains cationic and/or nonionic fabric softening agents.

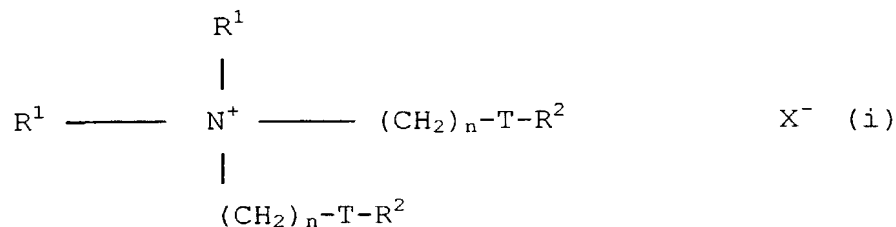
Types of cationic fabric softeners that may be used in the fabric softening compositions produced from the compositions of the invention include substantially water-insoluble quaternary ammonium materials, for example, a  
 5 compound having two C<sub>12</sub>-C<sub>22</sub> alkyl or alkenyl groups connected to a quaternary ammonium head group or a compound comprising a single long chain with an average chain length equal to or greater than C<sub>20</sub>. Preferably these are connected to the quaternary ammonium head group via at  
 10 least one ester link.

More preferably, the invention is useful for preparing compositions comprising quaternary ammonium material comprising a compound having two long chain alkyl or  
 15 alkenyl chains with an average chain length equal to or greater than C<sub>14</sub>. Even more preferably, each chain has an average chain length equal to or greater than C<sub>16</sub>. Most preferably at least 50% of each long chain alkyl or alkenyl group has a chain length of C<sub>18</sub>.

20

It is preferred if the long chain alkyl or alkenyl groups are predominantly linear. The especially preferred ester-linked quaternary ammonium materials for use in the invention can be represented by the formula (i):

25



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wherein each R<sup>1</sup> group is independently selected from C<sub>1-4</sub>

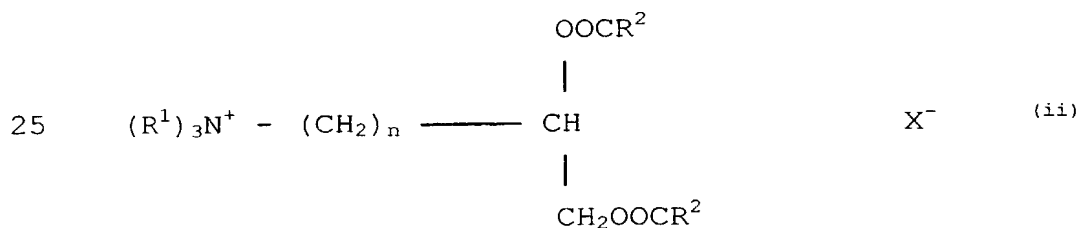
alkyl, hydroxyalkyl or C<sub>2-4</sub> alkenyl groups; and wherein each R<sup>2</sup> group is independently selected from C<sub>8-28</sub> alkyl or alkenyl groups; X<sup>-</sup> is any suitable counterion, for instance a halide, acetate or lower alkosulphate ion, such as chloride or methosulphate,

$$\begin{array}{ccc} \text{O} & & \text{O} \\ || & & || \\ \text{T} & \text{is } -\text{O}-\text{C}- & \text{or } -\text{C}-\text{O}-; \text{ and} \end{array}$$

10    n is an integer from 0-5

Di(tallowyloxyethyl) dimethyl ammonium chloride, available from Clariant, is especially preferred, also Di(hardened tallowyloxyethyl) dimethyl ammonium chloride, ex Clariant).  
 15 Dioleyl ester of methyl triethanol ammonium methosulphate wherein one R<sup>1</sup> is methyl and one is hydroxyethyl, both n are 2, both T are O-C=O, and both R<sup>2</sup> are tallow may also be used.

20 A second preferred type of quaternary ammonium material can be represented by the formula (ii):



wherein R<sup>1</sup>, n, R<sup>2</sup> and X<sup>-</sup> are as defined above.

30

It is advantageous for environmental reasons if the quaternary ammonium material is biologically degradable.

Preferred materials of this class such as 1,2 bis[hardened  
tallowoxyloxy]-3- trimethyl ammonium propane chloride and  
their method of preparation are, for example, described in  
US 4 137 180 (Lever Brothers). Preferably these materials  
5 comprise small amounts of the corresponding monoester as  
described in US 4 137 180 for example 1-hardened tallow-  
oxyloxy-2-hydroxy -3-trimethylammonium propane chloride.

The fabric softeners used in the compositions produced from  
10 the compositions of the invention are not required to meet  
the  $L\alpha$  to  $L\beta$  transition temperature referred to above. Only  
the cationic stabilisers, which may be of formulae (i) or  
(ii) above, are required to have the specified transition  
temperature. If compounds of these formulae are used only  
15 as a fabric softening active material, the transition  
temperature requirement does not apply.

Substantially water-insoluble fabric softening compounds  
are defined as fabric softening compounds having a  
20 solubility of less than  $1 \times 10^{-3}$  wt % in demineralised  
water at 20°C. Preferably the fabric softening compounds  
have a solubility of less than  $1 \times 10^{-4}$  wt%, more  
preferably less than  $1 \times 10^{-8}$  to  $1 \times 10^{-6}$  wt%.

25 The fabric softening compositions typically contain 1-8wt%  
of the fabric softening compound, and are known as dilute  
compositions. They may also contain higher amounts, such as  
8-50% softening compounds in which case they are known as  
concentrates.

Further ingredients, typically minor ingredients, may be added to the fabric softener compositions produced by the method described herein.

15 Example 1; concentrated perfume composition

	<u>grams of active</u> <u>ingredient</u>
Perfume* <sup>1</sup>	34.2
TETRANYL AO-1* <sup>2</sup>	10.0
Patent blue dye	2.0
(10% aqueous solution by weight)	

\*1 - SOFTLINE 2000 (ex Givaudan Roure; SOFTLINE IS A TRADEMARK)



\*2 - TETRANYL AO-1 is 90% dioleyl ester of methyl triethanol ammonium methosulphate, 10% IPA (Ex Kao). TETRANYL is a trademark.

5 Example 2; concentrated perfume composition

The composition below was prepared according to the method of example 1.

	<u>grams of active</u> <u>ingredient</u>
Perfume* <sup>3</sup>	34.2
AO-1* <sup>2</sup>	10.0
Patent blue dye (5% aqueous solution by weight)	2.0
*3 SOFTLINE B53 ex Givaudan Roure	

10 Example 3; concentrated perfume composition

The composition below was prepared according to the method of example 1.

	<u>grams of active</u> <u>ingredient</u>
Perfume* <sup>1</sup>	34.2
PRAPAGEN 3445* <sup>4</sup>	10.0
Patent blue dye (10% aqueous solution by weight)	2.0

- 15 \*4 - PRAPAGEN 3445 is 70% ditallowyl dimethyl quaternary ammonium chloride, 20%IPA and 10% water (ex Clariant). PRAPAGEN is a trademark.

Example 4; concentrated perfume composition

The composition below was prepared according to the method of example 1.

5

	<u>grams of active</u> <u>ingredient</u>
Perfume* <sup>5</sup>	34.2
ARQUAD 2T* <sup>6</sup> (as supplied)	12.2
RHODAMINE B dye (5% aqueous solution by weight)	2.0

\*<sup>6</sup> - cationic surfactant ditallowyl dimethyl quaternary ammonium chloride (ex Akzo). ARQUAD is a trademark.

\*<sup>5</sup> - HORIZON 2000 ex IFF. HORIZON is a trademark.  
RHODAMINE is a trademark.

10

Example 5; concentrated perfume composition

The composition below was prepared according to the method of example 1.

	<u>grams of active</u> <u>ingredient</u>
Perfume* <sup>5</sup>	34.2
AO-1 * <sup>2</sup>	10.0
Patent blue dye (10% aqueous solution by weight)	2.0

15

Example 6; concentrated perfume composition

The composition below was prepared according to the method of example 1.

	<u>grams of active</u> <u>ingredient</u>
Perfume <sup>*3</sup>	35.0
GENAPOL Coco 10 <sup>*7</sup>	3.0
AOT-1 <sup>*8</sup>	4.6
Patent blue dye (5% aqueous solution by weight)	2.0

<sup>\*7</sup> Genapol C-10 is coconut ethoxlated alcohol with an average of 10 moles of ethoxylate per mole of fatty alcohol (ex Clariant). GENAPOL is a trademark.

<sup>\*8</sup> TETRANYL AOT-1 is 80% dioleyl ester of methyl triethanol ammonium methosulphate, 20% dipropylene glycol solvent

10 Examples 1 to 6 were all stable, isotropic water-in-oil microemulsions.

Examples 7; use of example 1 to prepare a fabric softening composition

15

A fabric softening base composition comprising 94.5 parts water and 5 parts dihardened tallow dimethyl ammonium chloride was prepared. To this 0.5 parts of example 1 was added and the composition stirred until homogeneous (to provide approximately 0.37% perfume, 0.11% AO-1 and 0.002% dye). A stable fabric softening composition was produced that showed the same physical characteristics as the comparative example below.

20

A comparative example was produced by the conventional method of mixing together the following ingredients:

	<u>grams of active</u> <u>ingredient</u>
Dihardened tallow	5.0
dimethyl ammonium chloride	
Perfume* <sup>1</sup>	0.3
Patent blue dye	0.002
Water	to 100%

The fabric softening composition comparative example and  
5 the fabric softening composition produced from example 1  
both exhibited good stability on storage at room  
temperature.

#### Example 8

10 A concentrated fabric conditioner was prepared from 1,2  
bis[hardened tallowoyloxy]-3-trimethyl ammonium propane  
chloride following standard procedures but without perfume  
or dye. Subsequently the perfume dye mixture of example 2  
was added to this composition with gentle stirring. The  
15 Table below shows product viscosity, measured at  $106\text{S}^{-1}$ , as  
a measure of stability compared with a control product of  
identical composition but with perfume and dye added  
separately during the conditioner preparation.

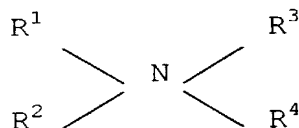


**Claims**

1. A liquid composition comprising;
  - (a) 15 - 95 wt% lipophilic perfume,
  - (b) 0.05 - 5 wt% water-soluble dye,
  - (c) 4 - 50 wt% of a stabilising agent comprising a cationic stabilising agent, and
  - (d) water miscible solventwherein the composition comprises between 0.1 to 20 wt% water, the cationic stabilising agent has an  $L\alpha$  to  $L\beta$  transition temperature of 45°C or below for a 5 wt% dispersion of the stabilising agent in water and the solvent is present in an amount of up to 10wt%.
2. A composition according to claim 1 wherein the composition is an isotropic liquid.
3. A composition according to claim 2 wherein the isotropic liquid is a water-in-oil microemulsion.
4. A composition according to any one of the preceding claims comprising 40-85 wt% perfume.
5. A composition according to any one of the preceding claims wherein the perfume has a solubility in water of equal to, or less than, 0.5g in 100 ml of water at 20°C.
6. A composition according to any one of the preceding claims comprising 0.2 wt% to 1 wt% dye.

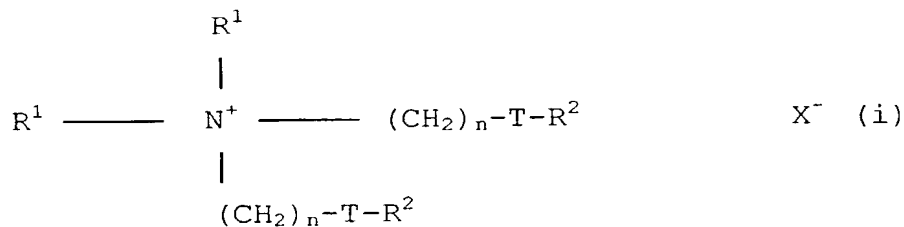
7. A composition according to any one of the preceding claims wherein the dye has a solubility in water of equal to or greater than, 5g in 100 ml of water at 20°C.
8. A composition according to any one of the preceding claims comprising 10 wt% - 30 wt% cationic surfactant as the stabilising agent.
9. A composition according to any one of the preceding claims wherein the cationic stabilising agent is a compound of general formula (A)

(A)



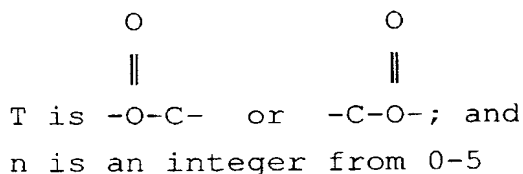
wherein  $R^1$  and  $R^2$  are independently  $C_1$ - $C_6$  alkyl, alkenyl, substituted alkyl or alkenyl groups, or hydroxyalkyl groups and  $R^3$  and  $R^4$  are independently  $C_8$ - $C_{28}$  alkyl, alkenyl, substituted alkyl or alkenyl groups, or hydroxalkyl groups

or, a compound of general formula (i)

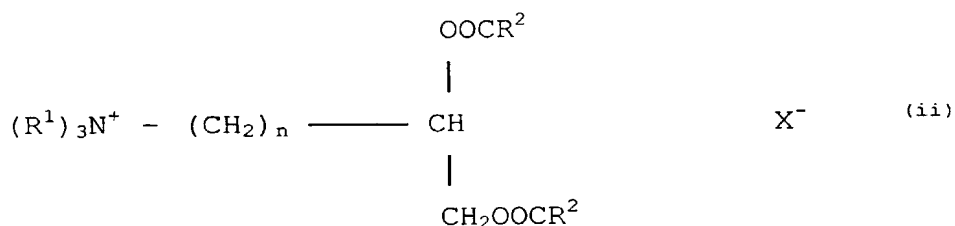


wherein each  $R^1$  group is independently selected from  $C_{1-4}$

alkyl, hydroxyalkyl or C<sub>2-4</sub> alkenyl groups; and wherein each R<sup>2</sup> group is independently selected from C<sub>8-28</sub> alkyl or alkenyl groups; X<sup>-</sup> is chloride or methosulphate.



or, a compound of general formula (ii)



wherein R<sup>1</sup>, n, R<sup>2</sup> and X<sup>-</sup> are as defined above.

10. A composition according to any one of the preceding claims wherein the weight ratio of perfume to dye is within the range 200:1 to 5:1, preferably 100:1 to 15:1.
11. A composition according to any one of the preceding claims wherein the weight ratio of perfume to stabilising agent is 10:1 to 1:1, preferably 5:1 to 1:1.
12. A composition according to any one of the preceding claims comprising 0.1- 10 wt% water.



13. A method of preparing a fabric softening composition comprising the steps;

(i) preparing a base composition comprising a cationic and/or nonionic fabric softening agent, and

(ii) adding to (i) a composition according to any one of the preceding claims, to produce the fabric softening composition.

14. A fabric softening composition obtainable by the method of claim 13.

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INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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<b>(21) International Application Number:</b> PCT/EP00/03724 <b>(22) International Filing Date:</b> 20 April 2000 (20.04.00) <b>(30) Priority Data:</b> 9910101.6 30 April 1999 (30.04.99) GB <b>(71) Applicant (for AE AU BB CA CY GB GD GH GM IE IL KE LC LK LS MN MW NZ SD SG SL SZ TT TZ UG ZA ZW only):</b> UNILEVER PLC [GB/GB]; Unilever House, Blackfriars, London EC4P 4BQ (GB). <b>(71) Applicant (for all designated States except AE AU BB CA CY GB GD GH GM IE IL IN KE LC LK LS MN MW NZ SD SG SL SZ TT TZ UG US ZA ZW):</b> UNILEVER NV [NL/NL]; Weena 455, NL-3013 AL Rotterdam (NL). <b>(71) Applicant (for IN only):</b> HINDUSTAN LEVER LIMITED [IN/IN]; Hindustan Lever House, 165/166 Backbay Reclamation, Maharashtra, Mumbai 400 020 (IN). <b>(72) Inventor; and</b> <b>(75) Inventor/Applicant (for US only):</b> FRASER, Stuart, Bernard [GB/GB]; Unilever Research Port Sunlight, Quarry Road East, Bebington, Wirral, Merseyside CH63 3JW (GB).		<b>(74) Agent:</b> HODGETTS, Catherine, Dawn; Unilever PLC, Patent Department, Colworth House, Sharnbrook, Bedford, Bedfordshire MK44 1LQ (GB). <b>(81) Designated States:</b> AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG). <b>Published</b> <i>With international search report.</i> <i>Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i>
<b>(54) Title:</b> CONCENTRATED PERFUME COMPOSITIONS AND MANUFACTURE OF FABRIC SOFTENING COMPOSITIONS THEREFROM <b>(57) Abstract</b> <p>The invention provides liquid compositions comprising: (a) 15 – 95 wt % lipophilic perfume, (b) 0.05 – 5 wt % water-soluble dye, (c) 4 – 50 wt % of a stabilising agent comprising a cationic stabilising agent, and (d) water miscible solvent wherein the composition comprises between 0.1 to 20 wt % water, the cationic stabilising agent has an <math>L\alpha</math> to <math>L\beta</math> transition temperature of 45 °C or less for a 5 wt % dispersion of the stabilising agent in water, and the solvent is present in an amount of up to 10 wt %. These concentrated perfume and dye compositions find particular application in fabric softening compositions. Also provided is a method of preparing a fabric conditioning composition by preparing a base composition comprising a cationic and/or nonionic fabric softening agent, and adding thereto, a composition of the invention. The fabric conditioning compositions thus produced are also provided. Simplified automated manufacture of fabric softening compositions is achieved.</p>		

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<b>CONCENTRATED PERFUME COMPOSITIONS AND MANUFACTURE OF FABRIC SOFTENING COMPOSITIONS THEREFROM</b>			
the specification of which (check only one item below):			
<input type="checkbox"/> is attached hereto.			
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I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.			
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UNITED KINGDOM	9910101.6	30 <sup>th</sup> April 1999	<input checked="" type="checkbox"/> YES <input type="checkbox"/> NO
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I hereby claim the benefit under Title 35, United States Code §120 of any United States application(s) or PCT international application(s) designating the United States of America that is/are listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in that /those prior application(s) in the manner provided by the first paragraph of Title 35, United States Code §112. I acknowledge the duty to disclose material information as defined in Title 37, Code of Federal Regulations §1.56 (a) which occurred between the filing date of the prior application(s) and the national or PCT international filing date of this application.

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201

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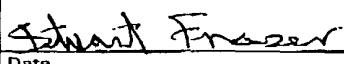
202

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203

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Signature of Inventor 201 	Signature of Inventor 202	Signature of Inventor 203
Date 30 <sup>th</sup> November 2001	Date	Date